

United States Patent [19]

Rigterink et al.

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[54] **SUBSTITUTED N-AROYL N'-PHENYL UREA COMPOUNDS**

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[21] Appl. No.: **520,033**

[22] Filed: **Aug. 3, 1983**

Related U.S. Application Data

[62] Division of Ser. No. 401,491, Jul. 26, 1982, Pat. No. 4,468,405.

[51] Int. Cl.³ **C07C 157/09; C07C 122/00; C07C 127/19**

[52] U.S. Cl. **260/453 AR; 260/454; 260/453 A; 564/26; 564/52; 564/442; 564/443; 564/440; 564/28; 564/29; 564/48; 564/49; 564/53; 564/54**

[58] Field of Search **260/454, 453 AR, 453 A; 564/26, 52, 442, 443, 440, 28, 29, 48, 49, 53, 54**

[56] References Cited

U.S. PATENT DOCUMENTS

4,284,628 8/1981 Nakagawa et al. 260/454

Primary Examiner—Henry R. Jiles

Assistant Examiner—Robert C. Whittenbaugh

[57] ABSTRACT

Dihalo and haloalkyl, haloalkoxy or haloalkylthio-substituted anilines and aniline derivatives, e.g. isocyanates that are useful in the preparation of acyl ureas having insecticidal activity.

5 Claims, No Drawings

SUBSTITUTED N-AROYL N'-PHENYL UREA COMPOUNDS

CROSS-REFERENCE TO RELATED APPLICATION

This is a divisional of application Ser. No. 401,491 filed July 26, 1982, now U.S. Pat. No. 4,468,405.

BACKGROUND OF THE INVENTION

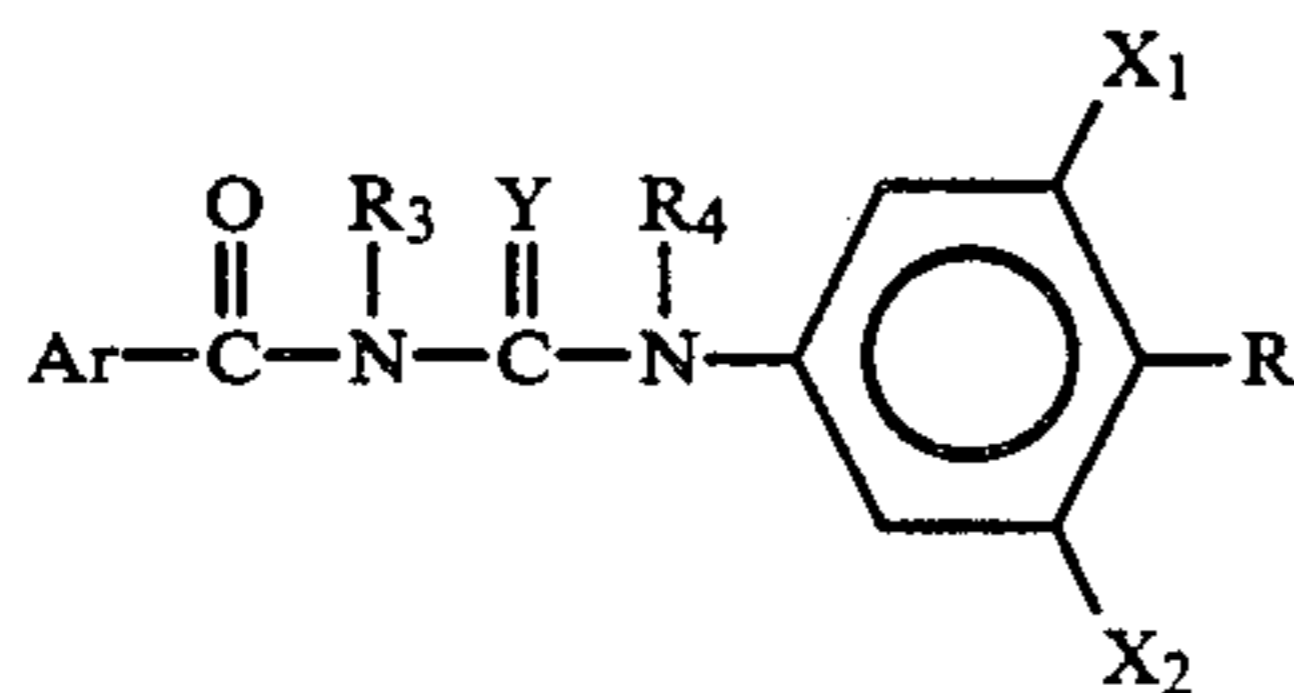
This invention relates to novel substituted N-aroyle N'-phenyl ureas, a process for producing them, insecticidal compositions containing them and a method for controlling certain insects.

Various insecticidal derivatives of urea are known in the art, such as, for example, U.S. Pat. Nos. 4,173,638; 4,005,223; 4,170,657; 4,139,636; 4,089,975 and German Patent Application No. 3,003,113.

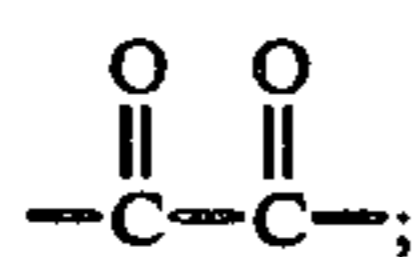
The N-aroyle N'-phenyl ureas of the present invention are more active and have a broader spectrum of effectiveness than the benzoylurea insecticides currently available.

SUMMARY OF THE INVENTION

The novel compounds of this invention are N-aroyle N'-phenyl ureas having the formula



wherein Ar is a substituted phenyl, pyridyl or pyrimidinyl radical wherein the substituents are chloro, bromo, fluoro, C₁-C₃ alkyl or C₁-C₃ alkoxy, with the proviso that at least one substituent is positioned ortho to the carbonyl group; R₃ and R₄ are individually H or C₁-C₄ alkyl or together they form the group



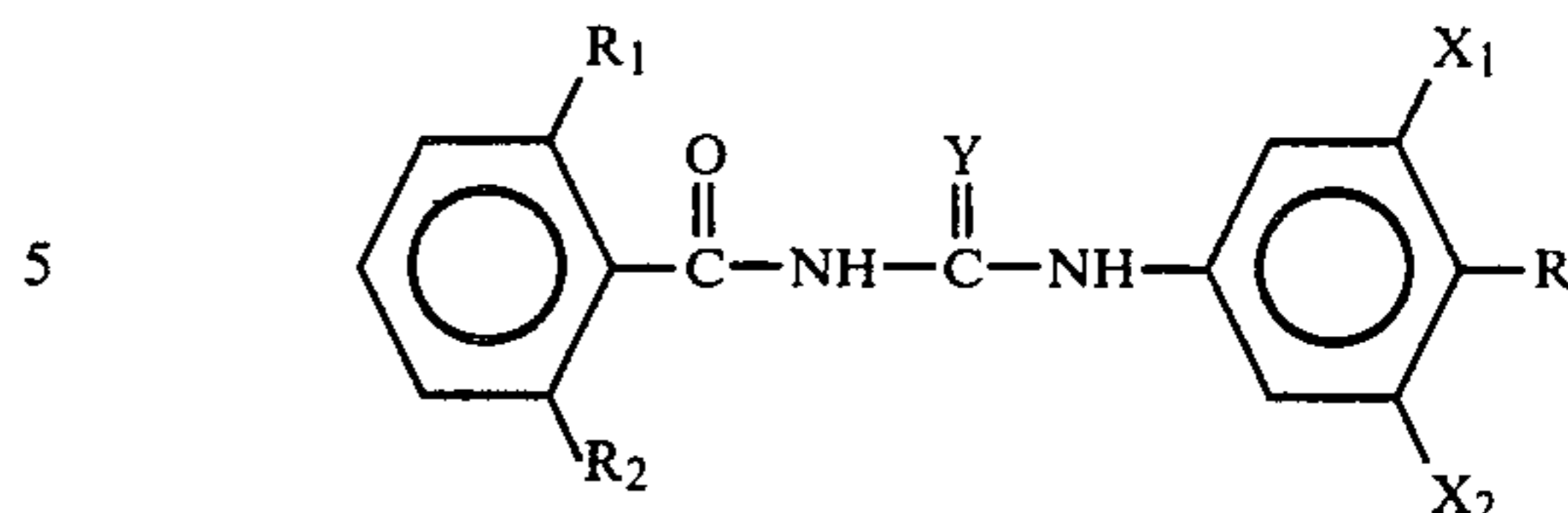
Y is O or S; X₁ and X₂ are halogen, and R is a C₁-C₄ haloalkyl, haloalkoxy or haloalkylthio group.

These novel compounds can be prepared by methods analogous to those known in the art, e.g., as taught in U.S. Pat. No. 4,139,636.

The invention also provides insecticidal compositions comprising an insecticidally effective amount of the above described N-aroyle N'-phenyl ureas in admixture with a suitable carrier or adjuvant therefore and a method for killing and/or controlling insects which comprises applying the active compound, alone or in admixture with a carrier, to the insects, the insect larvae or their habitats.

DETAILED DESCRIPTION OF THE INVENTION

The preferred compounds of this invention are those having the formula



where R₁ is F or Cl; R₂ is F, Cl or H; Y is O or S; X₁ and X₂ are halogen and R is OCF₃, OCF₂CHF₂, OCF₂CHClF, OCF₂CFHBr or OCF₂CHCl₂. Most preferably, R₁ is F or Cl R₂ is F, X₁ and X₂ are chlorine and R is OCF₂CHClF, OCF₂CHF₂ or OCF₂CHFBr.

The compounds of the present invention are normally crystalline solids of low solubility in water and of moderate solubility in many organic solvents. The compounds have low phytotoxicity and have exceptional activity in the control of various undesirable agricultural, household and veterinary insect pests.

Representative of the various insects which can be controlled by the active compounds of the present invention are members of the orders Lepidoptera, Coleoptera, Diptera, Orthoptera, Homoptera, Thysanoptera and Acarina. They are active against normally sensitive and resistant species at some stages of development. Examples of insect pests comprising the above include the tobacco budworm (*Heliothis virescens*), the beet armyworm (*Spodoptera exigua*), the Egyptian cotton leafworm (*Spodoptera littoralis*), the American bollworm (*Heliothis armigera*), the diamond-back moth (*Plutella maculipennis*), the gypsy moth (*Lymantria dispar*), the cutworm (*Agrotis segetum*), the Mediterranean flour moth (*Ephesia kuehniella*), the Colorado potato beetle (*Leptinotarsa decimlineata*), the mustard beetle (*Phaedon cochleariae*), the cotton boll weevil (*Anthonomus grandis*), the Mexican bean beetle (*Epilachna varivestis*), the khapra beetle (*Trogoderma granarium*), the housefly (*Musca domestica*), the lesser housefly (*Fannia canicularis*), the Mediterranean fruit fly (*Ceratitis capitata*), the black blow fly (*Phormia regina*), the cabbage rootfly (*Hylemya brassicae*), the yellow fever mosquito (*Aedes aegypti*), the malaria mosquito (*Anopheles stephensi*), the desert locust (*Schistocerca gregaria*), the migratory locust (*Locusta migratoria*), the German cockroach (*Blattella germanica*), the American cockroach (*Periplaneta americana*), the pear psylla (*Psylla pyricola*), the onion thrips (*Thrips tabaci*), and the citrus rust mite (*Phyllocoptruta oleivora*).

The compounds are highly active and can be employed to kill insects outright and/or to prevent adult emergence from juvenile forms of the insect. In such applications, the insect to be controlled and/or its habitat is contacted or treated with an insecticidal amount of one or more of the compounds of the present invention. The compounds may be administered orally to warm blooded animals from which they are excreted unchanged and they effectively combat the larvae of certain feces inhabiting insects, e.g., the face fly, horn fly and buffalo fly.

For all such uses, these compounds can be employed in unmodified form. However, the present invention embraces the use of an insecticidally-effective amount of the active ingredients in composition form with a material known in the art as an adjuvant or carrier.

Thus, for example, compositions employing one or a combination of these active ingredients can be in the form of a liquid or a dust; and the adjuvant employed

can be any one of a plurality of materials including aromatic solvents, petroleum distillates, water or other liquid carriers, propellant substances, surface-active dispersing agents, light absorbers and finely-divided carrier solids.

The exact concentration of one or a combination of the compounds of the present invention in a composition thereof with an adjuvant therefore can vary; it is only necessary that one or a combination of the compounds be present in a sufficient amount so as to make possible the application of an insecticidally-effective or inactivating dosage.

Generally, for practical applications, one or a combination of these active ingredients can be broadly applied to the insect larvae or their habitat in compositions containing from about 0.0001 to about 98 percent by weight, preferably 5 to 50 percent by weight, of the compounds.

The invention is further illustrated by the following examples.

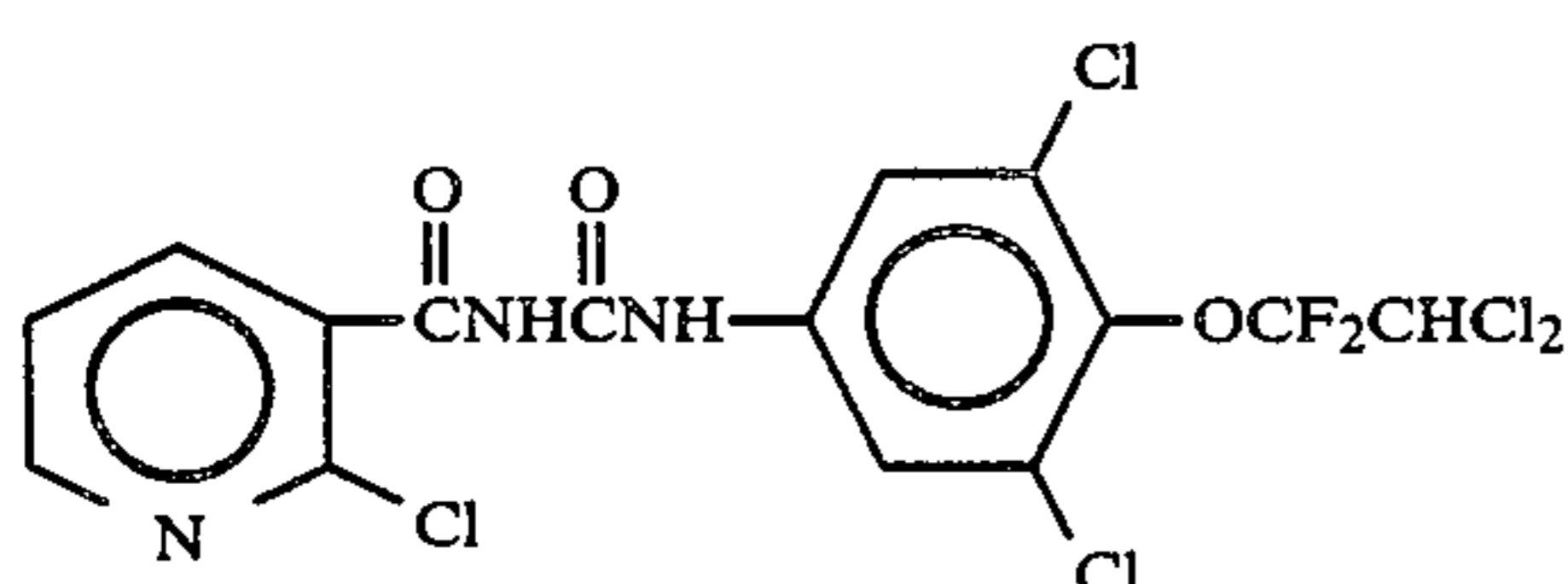
EXAMPLE 1

3,5-Dichloro-4-(2,2-dichloro-1,1-difluoroethoxy)phenyl isocyanate

3,5-Dichloro-4-(2,2-dichloro-1,1-difluoroethoxy)benzenamine (12 g, 0.04 mole) was added to a solution of 20 g oxalyl chloride in 100 ml CCl₄ and heated under reflux with stirring for 0.5 hour. A solid precipitated. The carbon tetrachloride and excess oxalyl chloride were removed by evaporation in a rotary evaporator. The residue (16 g) was a slightly oily white solid, which was used in the next reaction without purification.

EXAMPLE 2

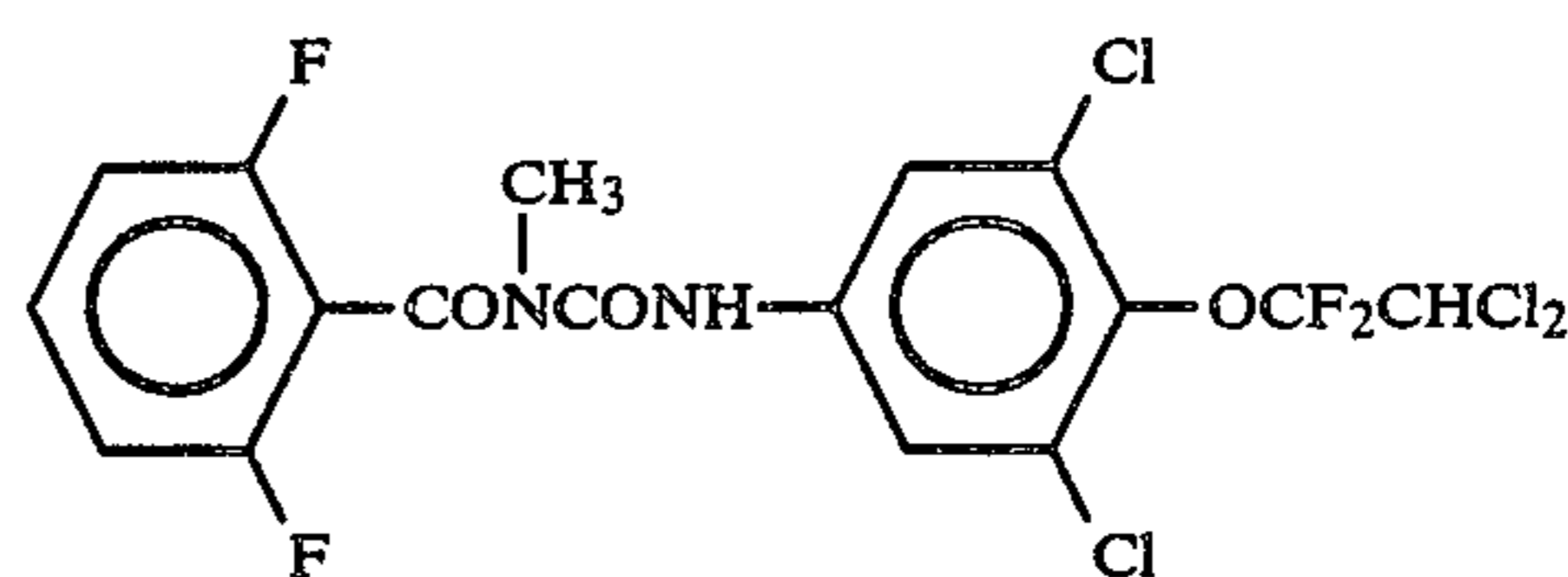
N-(((3,5-Dichloro-4-(2,2-dichloro-1,1-difluoroethoxy)phenyl)amino)carbonyl)-2-chloro-3-pyridinecarboxamide



2-Chloro-3-pyridinecarboxamide (6.3 g, 0.04 mole) and 3,5-dichloro-4-(2,2-dichloro-1,1-difluoroethoxy)phenyl isocyanate (crude; 16 g, 0.04 mole) were added to 250 ml xylene and heated under reflux with stirring for two hours. After cooling in ice water, the precipitated product was removed by suction filtration. This was recovered as 2-chloro-3-pyridine carboxamide. A second crop was obtained by adding about 400 ml hexane to the filtrate. The second crop was a mixture of the desired material and recovered amide. The desired material was isolated and purified by recrystallizing twice from toluene. M.P. = 189°-192° C. NMR spectral features supported the structural assignment.

EXAMPLE 3

N-(((3,5-Dichloro-4-(2,2-dichloro-1,1-difluoroethoxy)phenyl)amino)carbonyl)-2,6-difluoro-N-methylbenzamide

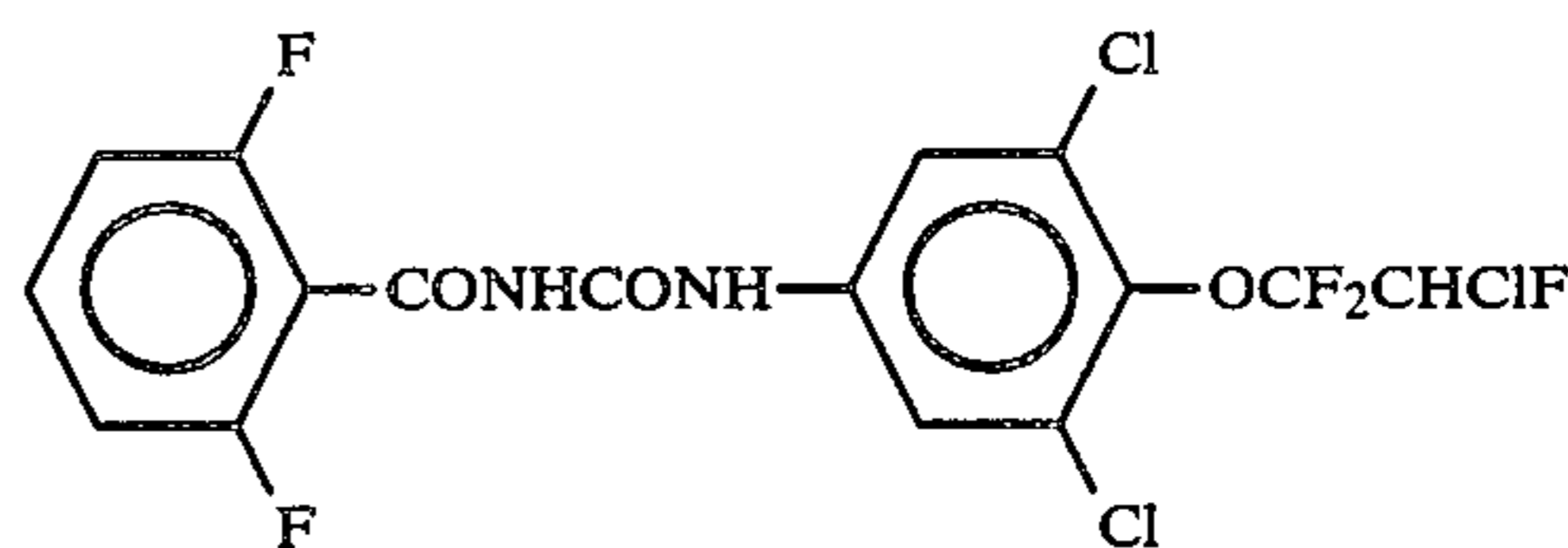


N-(((3,5-Dichloro-4-(2,2-dichloro-1,1-difluoroethoxy)phenyl)amino)carbonyl)-2,6-difluorobenzamide (9.9 g, 0.02 mole) and KOH (1.4 g, 87 percent purity, 0.02 mole) were added to 50 ml dimethylformamide with stirring, giving a clear solution. Iodomethane (3.2 g, 0.022 mole) was added and the resulting solution was stirred at room temperature for about 24 hours. The precipitated product was collected by suction filtration and dried. The product was purified by recrystallization from isopropanol. The purified product was a white solid melting at 153°-155° C. NMR spectral features support the structural assignment.

Elemental Analysis: Calcd: C, 40.18; H, 1.98; N, 5.51. Found: C, 40.3; H, 2.10; N, 5.43.

EXAMPLE 4

N-(((3,5-Dichloro-4-(2-chloro-1,1,2-trifluoroethoxy)phenyl)amino)carbonyl)-2,6-difluorobenzamide

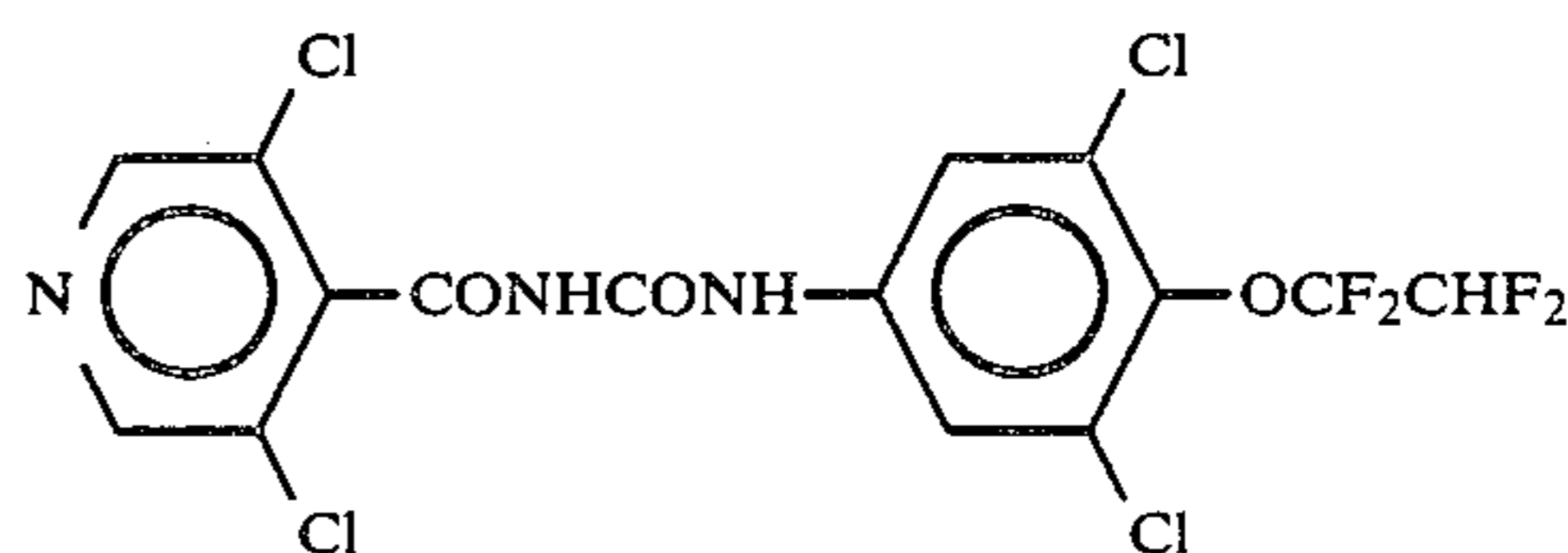


2,6-Difluorobenzoyl isocyanate (5.5 g, 0.03 mole) was added to a solution of 3,5-dichloro-4-(2-chloro-1,1,2-trifluoroethoxy)benzenamine (8.8 g, 0.03 mole) in 150 ml toluene and heated under reflux with stirring for one hour giving a clear solution. Toluene was removed by evaporation in a rotary evaporator. The residue was recrystallized from aqueous acetic acid and then from acetonitrile giving a white solid (8.3 g, 58 percent yield), melting at 178°-180° C.

Elemental analysis: Calcd: C, 40.23; H, 1.69; N, 5.87. Found: C, 40.20; H, 1.79; N, 5.83.

Employing the above procedures and appropriate starting materials, the following compounds were prepared:

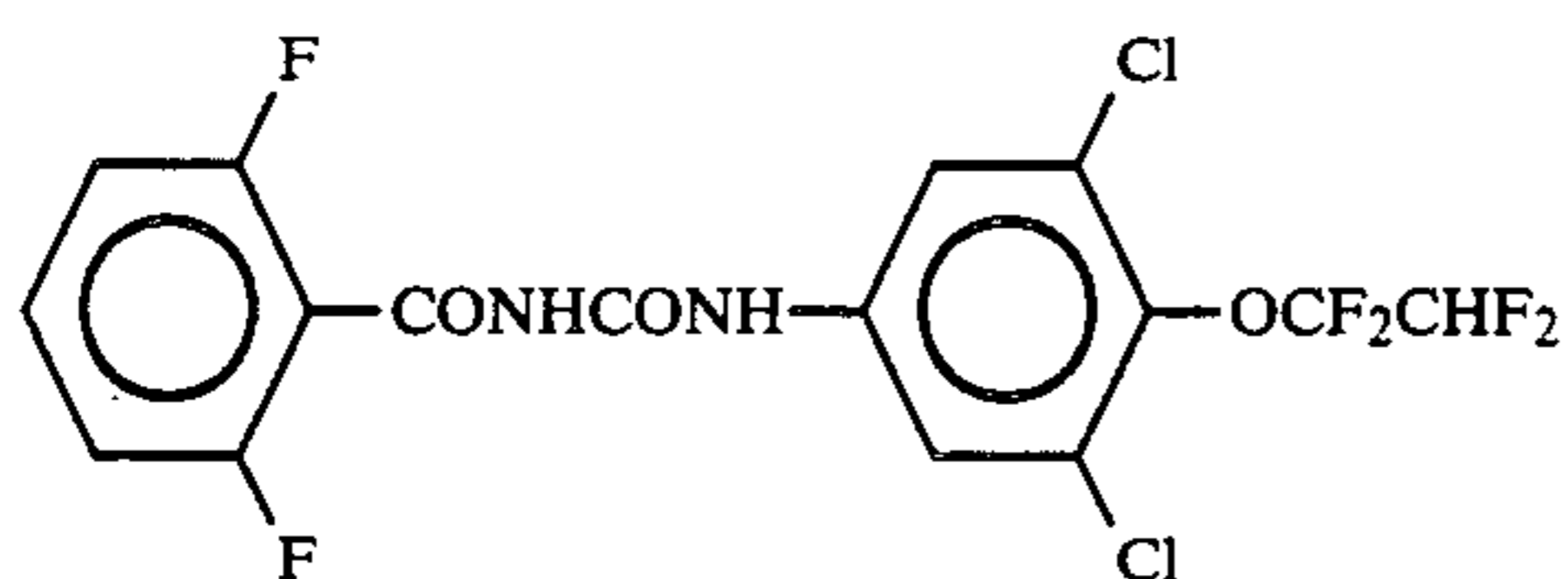
N-(((3,5-Dichloro-4-(1,1,2,2-tetrafluoroethoxy)phenyl)amino)carbonyl)-3,5-dichloro-4-pyridinecarboxamide.



M.P. = 217°-219° C.

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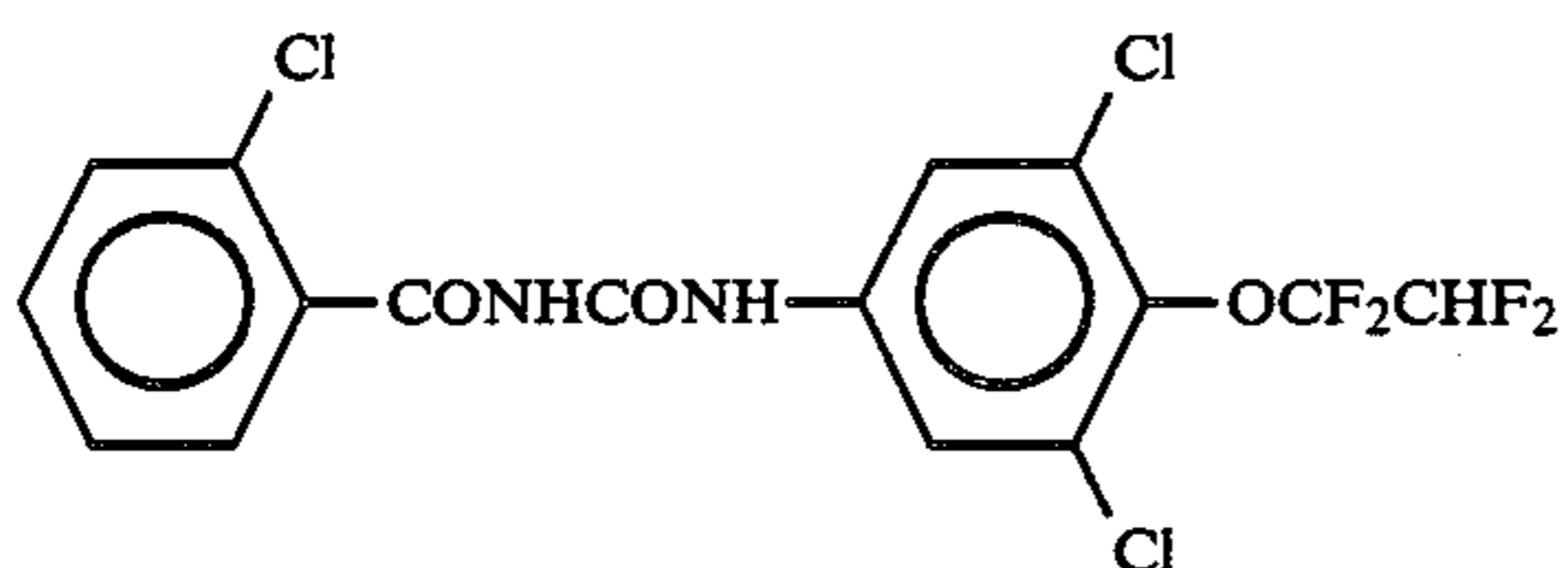
N-(((3,5-dichloro-4-(1,1,2,2-tetrafluoroethoxy)phenyl)amino)carbonyl)-2,6-difluorobenzamide.



M.P. = 197°-199° C.

Analysis: Calcd: C, 41.67; H, 1.75; N, 6.08. Found: C, 41.8; H, 1.92; N, 6.06.

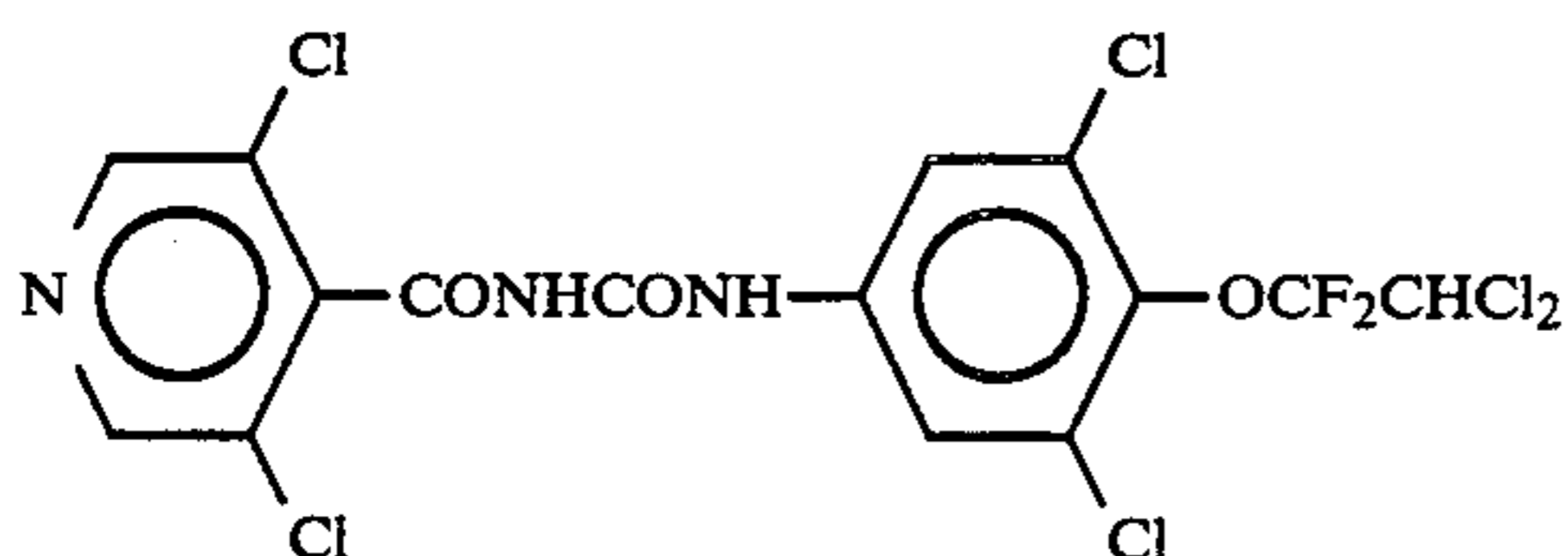
N-(((3,5-dichloro-4-(1,1,2,2-tetrafluoroethoxy)phenyl)amine)carbonyl)-2-chlorobenzamide.



M.P. = 173°-175° C.

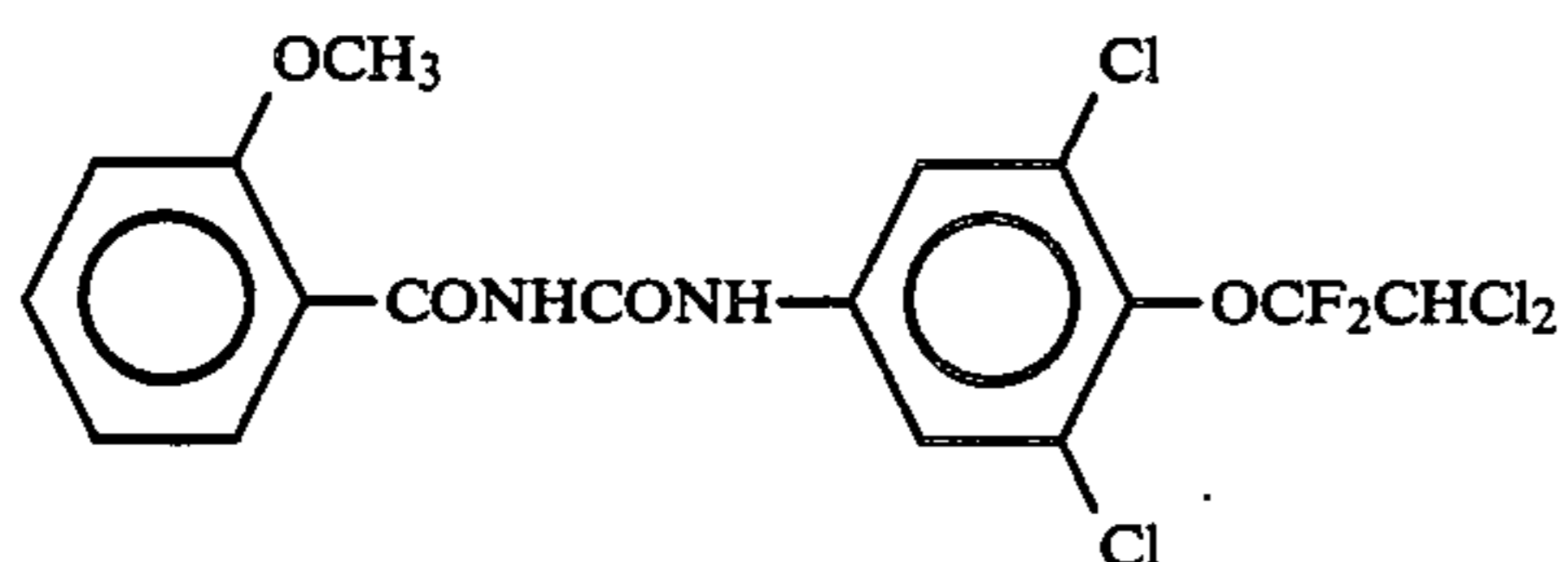
Analysis: Calcd: N, 6.10. Found: N, 6.34.

3,5-Dichloro-N-(((3,5-dichloro-4-(2,2-dichloro-1,1-difluoroethoxy)phenyl)amino)carbonyl)-4-pyridinecarboxamide.



M.P. = 228°-230° C.

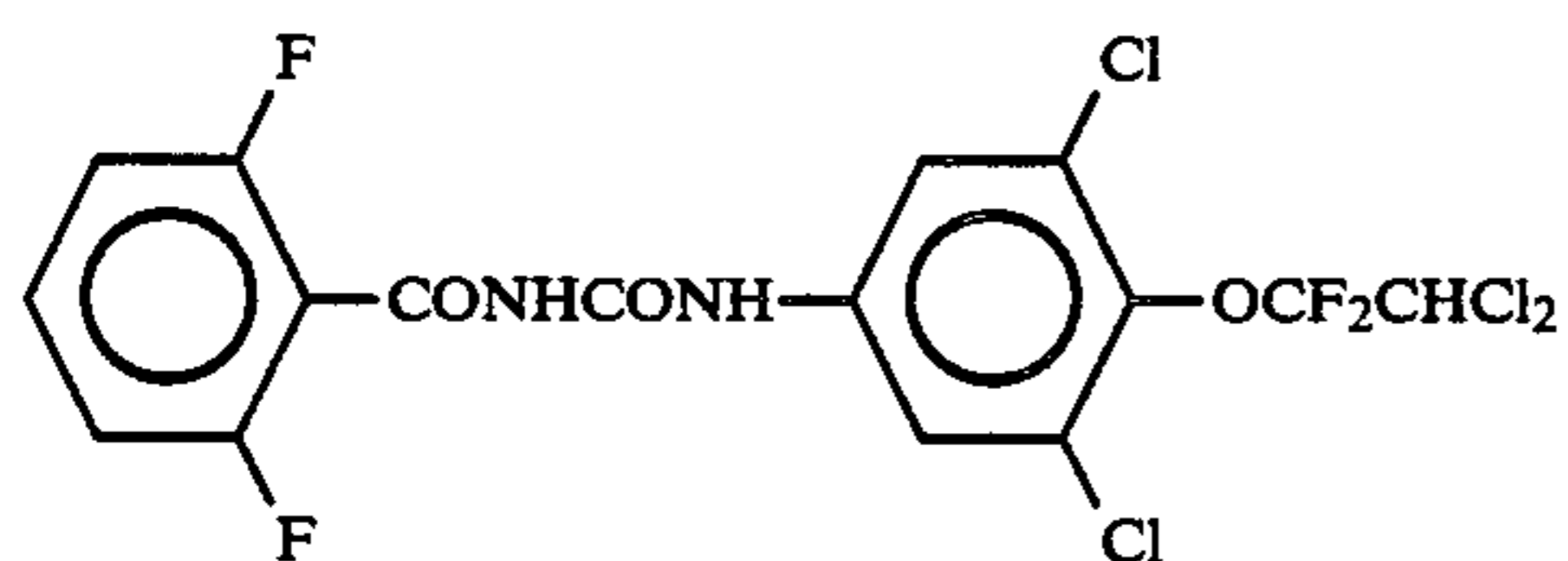
N-(((3,5-Dichloro-4-(2,2-dichloro-1,1-difluoroethoxy)phenyl)amino)carbonyl)-2-methoxybenzamide.



M.P. = 156°-158° C.

Analysis: Calcd: 41.83% C; 2.48% H; 5.74% N. Found: 41.9% C; 2.57% H; 5.92% N.

N-(((3,5-Dichloro-4-(2,2-dichloro-1,1-difluoroethoxy)phenyl)amino)carboxyl)-2,6-difluorobenzamide.



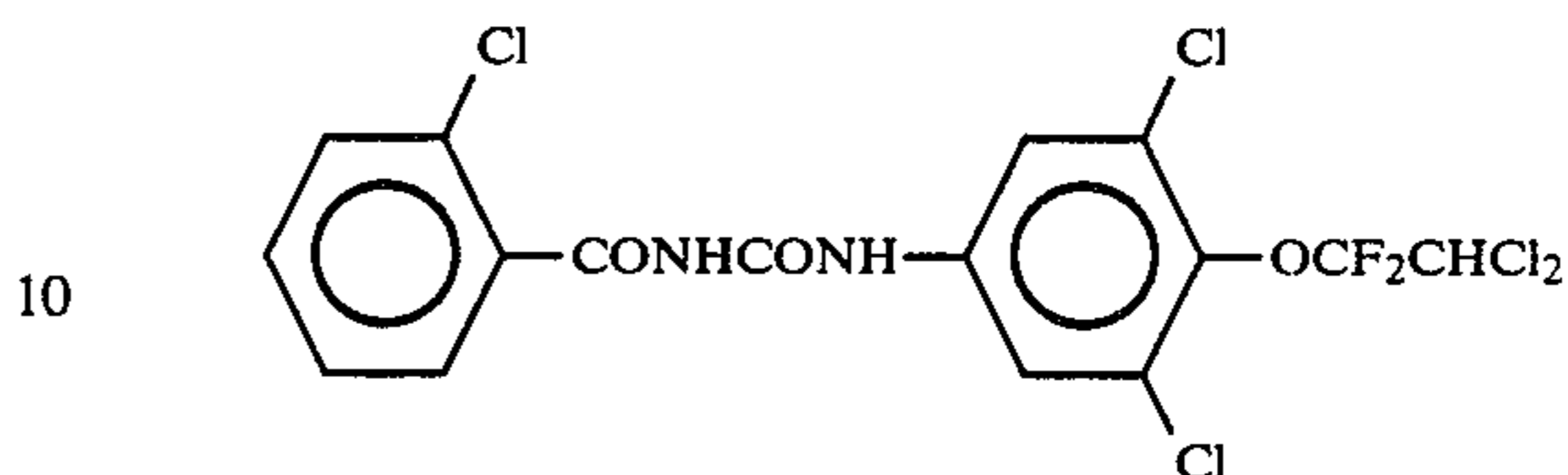
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M.P. = 199°-201° C.

Analysis: Calcd: N, 5.67. Found: N, 5.66.

2-Chloro-N-(((3,5-dichloro-4-(2,2-dichloro-1,1-difluoroethoxy)phenyl)amino)carbonyl)benzamide.

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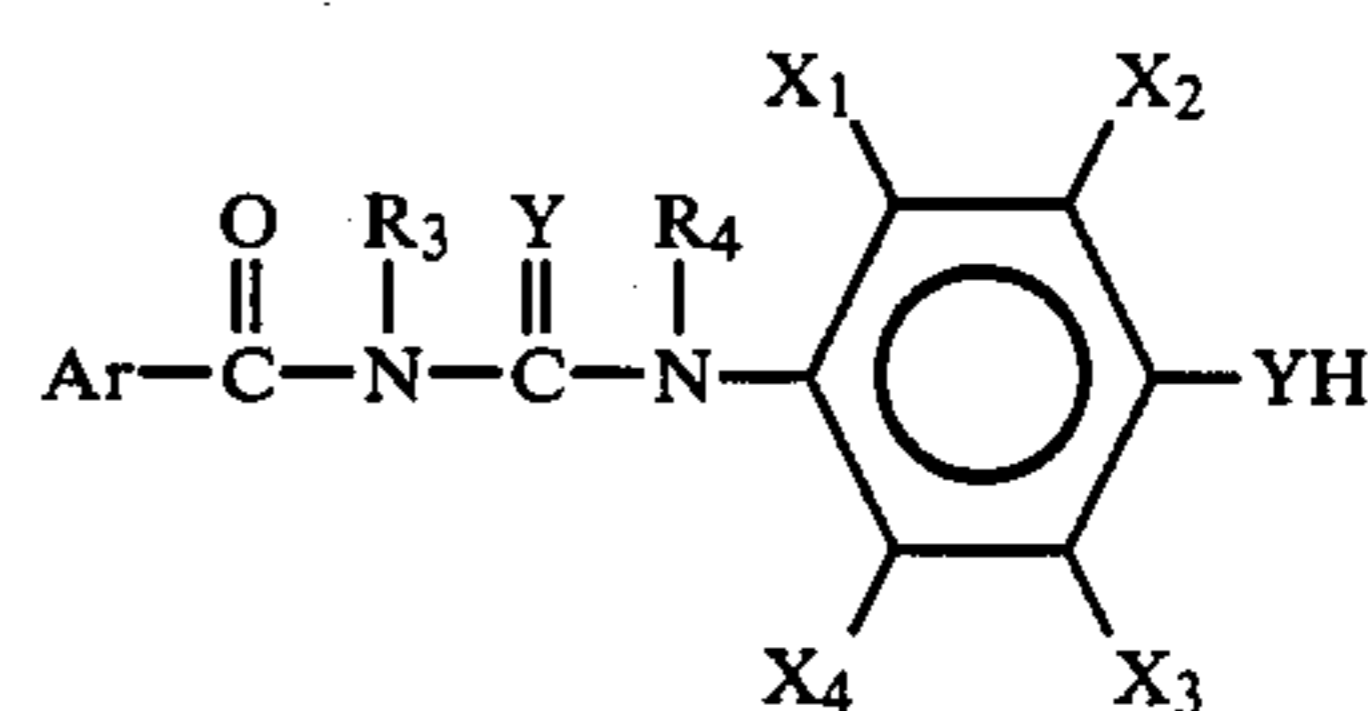
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M.P. = 200°-202° C.

Analysis: Calcd: N, 5.69. Found: N, 5.60.

In addition to the above described preparative methods, many of the compounds of this invention can be made by reacting a compound having the formula

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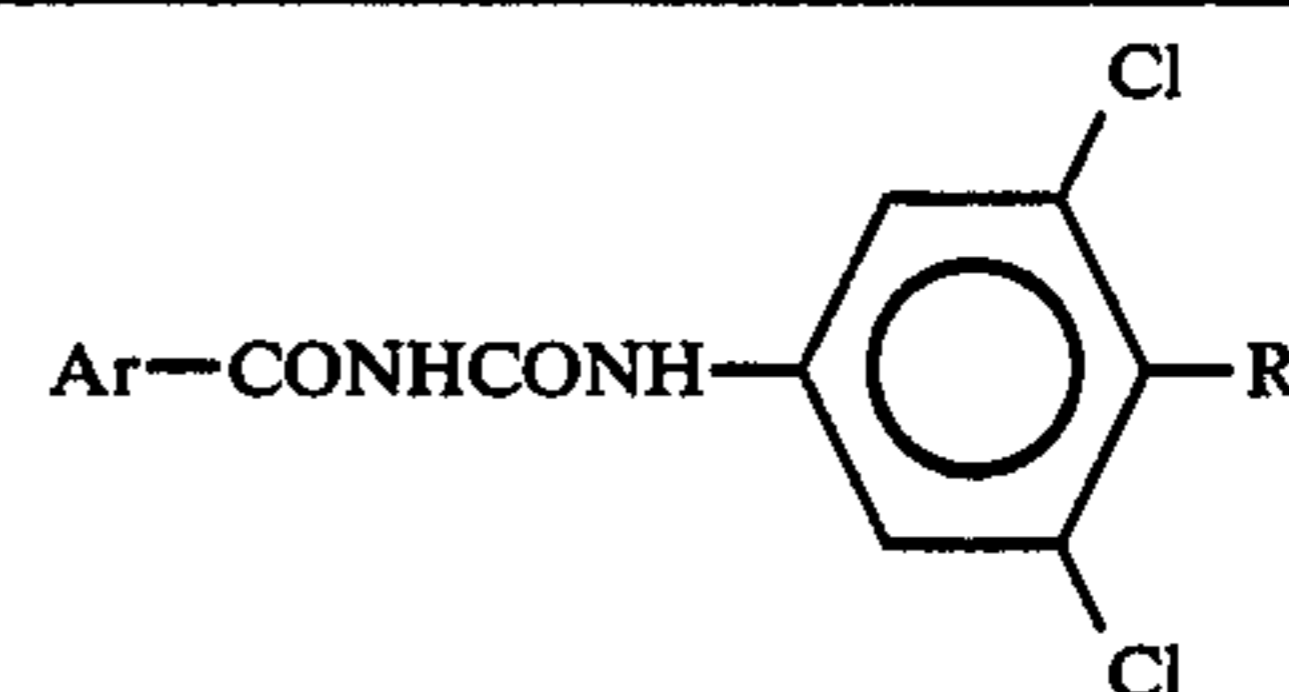
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wherein all of the substituents are as above defined, with a haloalkene as is known in the art.

The biological activity of several of these compounds was determined. In the beet armyworm test, cotton leaves were dipped in aqueous suspensions of the chemicals, dried, excised and placed into petri dishes with five second-instar beet armyworm (*Spodoptera exigua*) larvae. Mortality counts were made five days later. The tobacco budworm test was the same except that five tobacco budworm (*Heliothis virescens*) larvae were placed onto the treated leaves. The results are summarized below:

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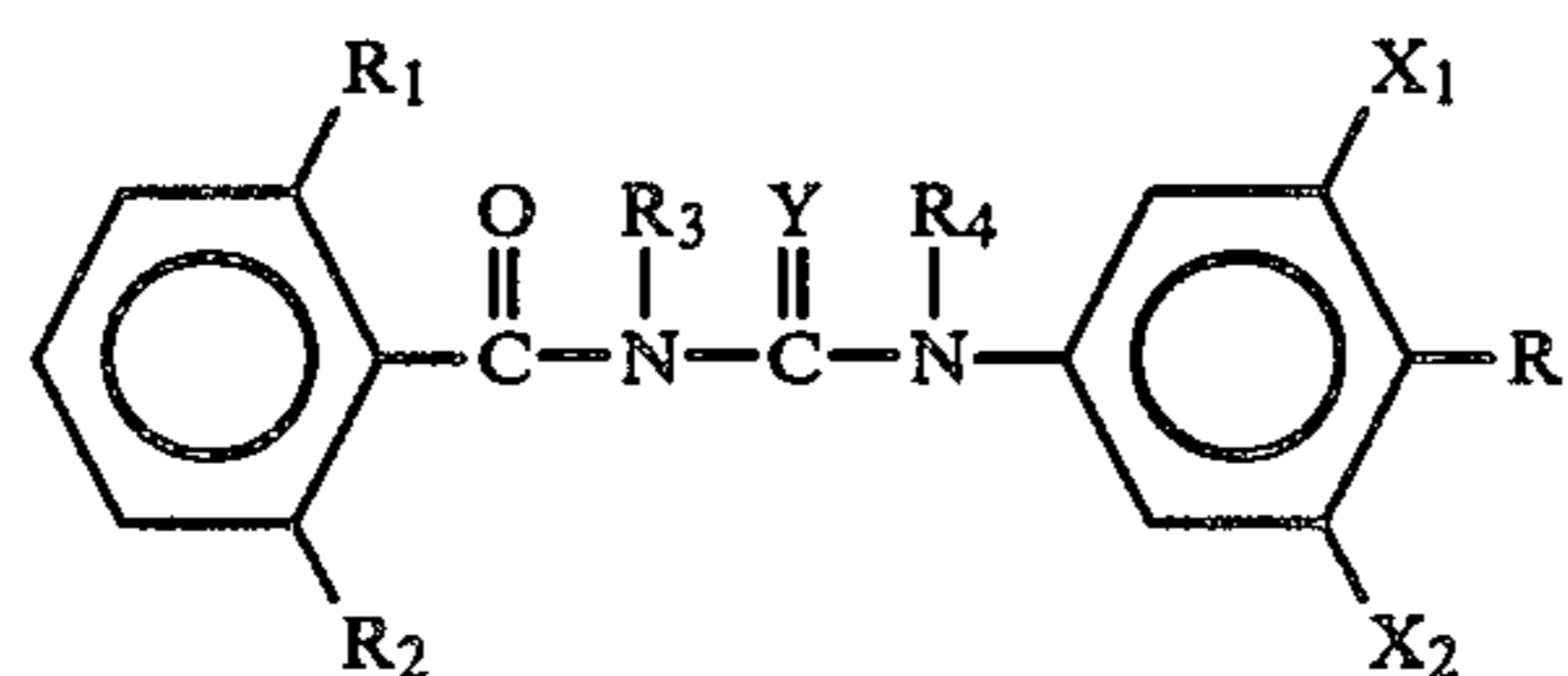
Compound	Ar	Percent Control at Indicated Dosage, ppm					
		Beet Army Worm Test (1)			Tobacco Budworm Test (2)		
		400	100	25	400	100	25
A	2,6-difluorophenyl	100	100	100	100	100	80
B	2-chlorophenyl	100	100	100	100	80	40

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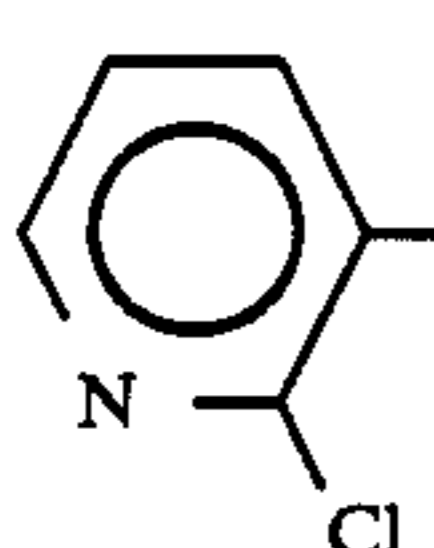
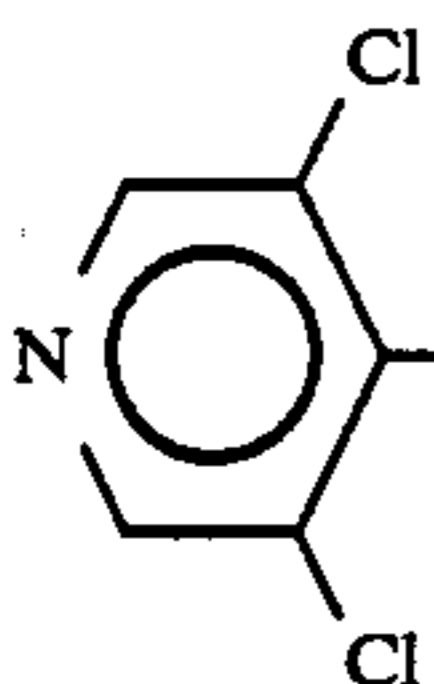
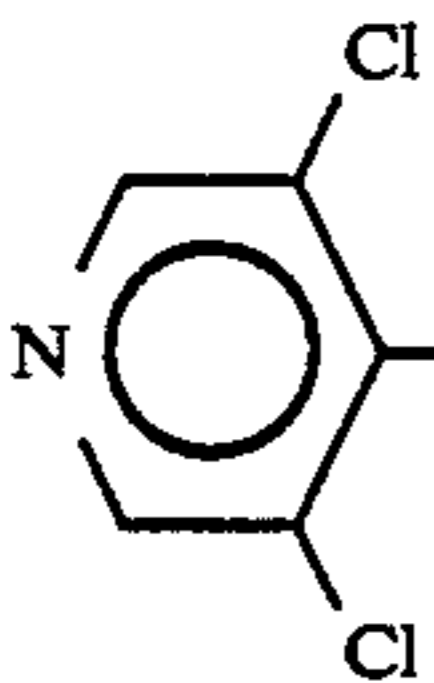
(1) Untreated check mortality 0%.
(2) Untreated check mortality 27%.

Employing the above described preparative and testing methods, the compounds listed in the following table were prepared and tested. The test results, LD₉₀ ppm, indicate the dosage necessary to obtain 90 percent kill of the indicated insect.

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Ex. No.	R ₁	R ₂	R ₃	R ₄	Y	X ₁	X ₂	R	Melting Point °C.	LD ₉₀ , ppm	
										Beet Army-worm	Tobacco Budworm
1	F	F	H	H	O	Cl	Cl	OCF ₂ CHF ₂	204-206	1.6	1.6
2	F	F	H	H	O	Cl	Cl	OCF ₂ CHFCl	176-178	1.2	1.2
3	F	F	H	H	O	Cl	Cl	OCF ₂ CHFBBr	170-172	1.2	2.5
4	F	F	H	H	O	Cl	Cl	OCF ₂ CHF ₂	170-172		12.5
5	F	F	H	H	O	Cl	Cl	OCF ₂ CHCl ₂	180-182	5.0	25.0
6	F	F	H	H	O	Cl	Cl	OCF ₂ CH ₂ Cl	188-190	100.0	100.0
7	F	F	H	H	O	Cl	Cl	OCF ₂ CHBr ₂	215-217	25.0	400.0
8	F	F	H	H	O	Cl	Cl	OCFClCHFCI	203-205	1.25	25.0
9	F	F	H	H	O	Cl	Cl	OCF ₃	218-220	0.6	12.5
10	F	F	H	H	O	Cl	Cl	OCF ₂ CF ₃	224-227	12.5	50.0
11	F	F	H	H	O	Cl	Cl	SCF ₂ CHFCl	250-260	200.0	>400.0
12	F	F	H	H	O	Cl	Cl	SCF ₂ CHF ₂	166-168	3.1	12.5
13	Cl	H	H	H	O	Cl	Cl	OCF ₂ CHFCl	178-180	5.0	6.2
14	Cl	H	H	H	O	Cl	Cl	OCF ₂ CHF ₂	173-175	100.0	25.0
15	Cl	H	H	H	O	Cl	Cl	OCF ₂ CHFBBr	183-185	2.5	10.0
16	Cl	H	H	H	O	Cl	Cl	OCF ₂ CHF ₂	171-174		50.0
17	Cl	H	H	H	O	Cl	Cl	OCF ₂ CHCl ₂	162-164	6.2	200.0
18	Cl	H	H	H	O	Cl	Cl	OCF ₂ CHBr ₂	217-219	25.0	400.0
19	Cl	H	H	H	O	Cl	Cl	OCF ₃	203-205	2.5	12.5
20	Cl	H	H	H	S	Cl	Cl	OCF ₂ CHFCl	137-139	10.0	200.0
21	Cl	H	H	H	S	Cl	Cl	OCF ₂ CHF ₂	140-142	400.0	50.0
22	F	F	H	H	S	Cl	Cl	OCF ₂ CHFCl	149-151	12.5	12.5
23	F	F	H	H	S	Cl	Cl	OCF ₂ CHF ₂			
24	F	H	H	H	O	Cl	Cl	SCF ₂ CHFCl	245-250	>400.0	>400.0
25	F	H	H	H	O	Cl	Cl	OCF ₂ CHFCl	165-167	100.0	25.0
26	F	Cl	H	H	O	Cl	Cl	OCF ₂ CHF ₂			
27	F	Cl	H	H	O	Cl	Cl	OCF ₂ CHFCl	205-207	2.5	5.0
28	F	Cl	H	H	O	Cl	Cl	OCF ₂ CHFBBr	184-186	2.5	5.0
29	Cl	Cl	H	H	O	Cl	Cl	OCF ₂ CHF ₂	217-219	25.0	25.0
30	Cl	Cl	H	H	O	Cl	Cl	OCF ₂ CHFCl	221-223	6.2	25.0
31	Cl	Cl	H	H	O	Cl	Cl	OCF ₂ CHFBBr	200-202	100.0	12.5
32	Cl	Cl	H	H	O	Cl	Cl	SCF ₂ CHF ₂	213-215	50.0	200.0
33	F	F	CH ₃	H	O	Cl	Cl	OCF ₂ CHCl ₂	153-155	100.0	>400.0
34	F	F	CH ₃	H	O	Cl	Cl	OCF ₂ CHFCl	128-130	100.0	50.0
35	F	F	CH ₃	H	O	Cl	Cl	OCF ₂ CHF ₂	102-104	100.0	50.0
36	F	F			O	Cl	Cl	OCF ₂ CHCl ₂	194-197	400.0	>400.0
37	F	F			O	Cl	Cl	OCF ₂ CHF ₂	166-169	100.0	>400.0
38	F	F			O	Cl	Cl	OCF ₂ CHFCl	190-192	400.0	400.0
39	F	F	H	H	O	Br	Br	OCF ₂ CHF ₂	201-203	3.7	10.0
40	CH ₃	H	H	H	O	Cl	Cl	OCF ₂ CHFCl	163-165		>200.0
41	CH ₃	CH ₃	H	H	O	Cl	Cl	OCF ₂ CHFCl	175-177		200.0
42	C ₂ H ₅	C ₂ H ₅	H	H	O	Cl	Cl	OCF ₂ CHFCl	182-184	100.0	400.0
43	OCH ₃	H	H	H	O	Cl	Cl	OCF ₂ CHCl ₂	156-158	>400.0	>400.0
44	OCH ₃	OCH ₃	H	H	O	Cl	Cl	OCF ₂ CHFCl	203-206	100.0	25.0
45	OCH(CH ₃) ₂	H	H	H	O	Cl	Cl	OCF ₂ CHFCl	239-241	>400.0	>400.0
46	OCH(CH ₃) ₂	H	H	H	O	Cl	Cl	OCF ₂ CHF ₂	242-244	>400.0	>400.0

Ar	R	M.P., °C.	LD ₉₀ , ppm	
			Beet Armyworm	Tobacco Budworm
			Cl	
$\text{Ar}-\overset{\text{O}}{\parallel}{\text{C}}-\text{NH}-\overset{\text{O}}{\parallel}{\text{C}}-\text{NH}$				
	OCF ₂ CHCl ₂	189-192	100	>400
	OCF ₂ CHF ₂	217-219	>400	>400
	OCF ₂ CHCl ₂	228-230	>400	>400
				

In further embodiments, the compounds of the present invention or compositions containing the same, can be advantageously employed in combination with one or more additional pesticidal compounds. Such additional pesticidal compounds may be insecticides, nematocides, acaricides, herbicides, fungicides or bactericides that are compatible with the compounds of the present invention in the medium selected for application and not antagonistic to the activity of the present compounds. Accordingly, in such embodiments the pesticidal compound is employed as a supplemental toxicant for the same or for a different pesticidal use, or as an additament. The compounds in combination can generally be present in the ratio of from 1 to 100 parts of the

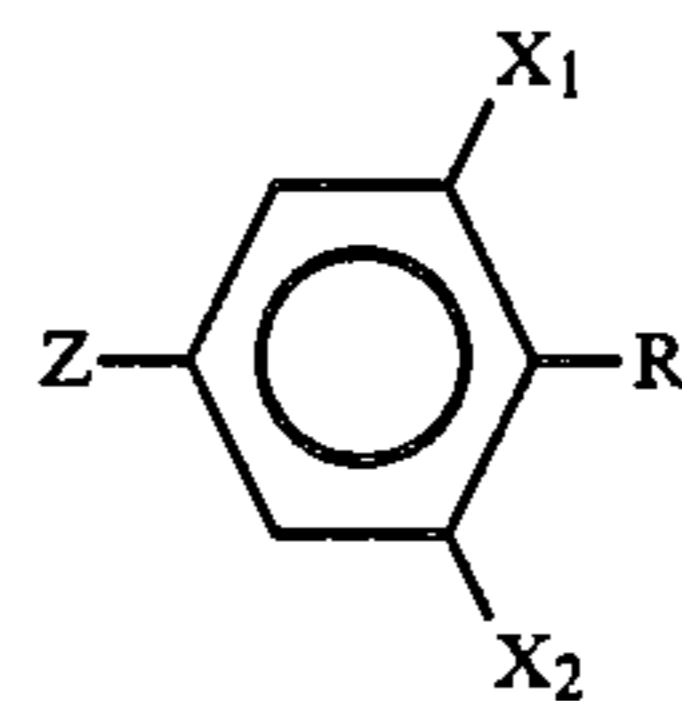
compound of the present invention with from 100 to 1 parts of the additional compound(s).

The compounds of this invention are, or tend to be, slow acting, i.e., they disrupt the molting of the insect, thereby killing it. As a result, some time can pass before the insects are killed. Accordingly, an increased benefit can be obtained by combining the compounds of this invention with quicker acting insecticides such as, for example organophosphorus compounds, carbamates and pyrethroids. Because of this different mode of action, the compounds of this invention kill or control insects which have, or may be developing, resistance to the more common insecticides and thus they inhibit or delay the development of resistance to such insecticides.

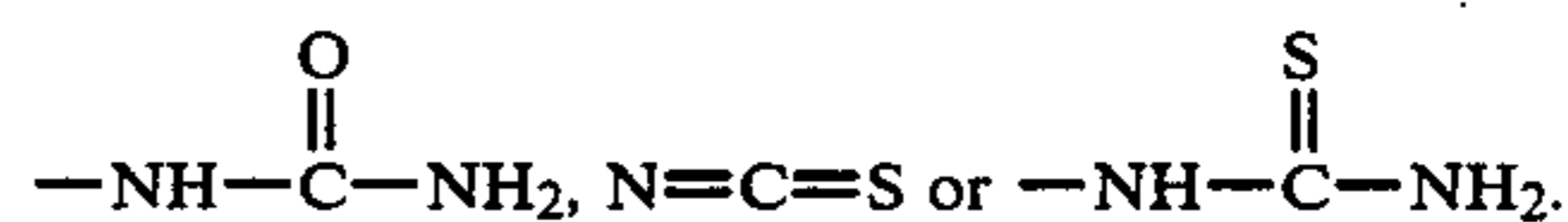
Various modifications may be made in the present invention without departing from the spirit or scope thereof and it is understood that we limit ourselves only as defined in the appended claims.

We claim:

1. A compound having the formula



wherein X₁ and X₂ are halogen which may be the same or different, R is a C₁-C₄ haloloweralkyl, haloloweralkoxy or haloloweralkylthio group and Z is NH₂, —N=C=O,



2. Compound of claim 1 wherein X₁ and X₂ are independently F, Cl or Br and R is OCF₃, OCF₂CHCl₂, OCF₂CHF₂, OCF₂CHFCl or OCF₂CHFBr.

3. Compound of claim 2 wherein X₁ and X₂ are both Cl and R is OCF₂CHF₂.

4. Compound of claim 2 wherein X₁ and X₂ are both Cl and R is OCF₂CHFCl.

5. Compound of claim 2 wherein X₁ and X₂ are both Cl and R is OCF₂CHFBr.

* * * * *

REEXAMINATION CERTIFICATE (905th)
United States Patent [19] [11] **B1 4,536,341**
Rigterink et al. [45] **Certificate Issued Jul. 26, 1988**

[54] **SUBSTITUTED N-AROYL N-PHENYL UREA COMPOUNDS**

[75] **Inventors: Raymond H. Rigterink, Midland, Mich.; Ronald J. Sbragia, Clayton, Calif.**

[73] **Assignee: The Dow Chemical Company, Midland, Mich.**

Reexamination Request:
No. 90/001,143, Dec. 22, 1986

Reexamination Certificate for:
Patent No.: **4,536,341**
Issued: **Aug. 20, 1985**
Appl. No.: **520,033**
Filed: **Aug. 3, 1983**

Related U.S. Application Data

- [62] Division of Ser. No. 401,491, Jul. 26, 1982, Pat. No. 4,468,405.
- [51] **Int. Cl.⁴ C07C 119/048; C07C 127/22; C07C 153/057; C07C 157/12**
- [52] **U.S. Cl. 558/13; 560/358; 564/23; 564/26; 564/28; 564/29; 564/44; 564/48; 564/49; 564/52; 564/53; 564/54; 564/440; 564/442; 564/443**
- [58] **Field of Search 560/358; 558/13; 564/23, 26, 28, 29, 44, 48, 49, 53, 52, 54, 440, 442, 443**

[56] **References Cited**
FOREIGN PATENT DOCUMENTS

762485 7/1967 Canada 564/442

Primary Examiner—Alan Rotman

[57] **ABSTRACT**

Dihalo and haloalkyl, haloalkoxy or haloalkylthio-substituted anilines and aniline derivatives, e.g. isocyanates that are useful in the preparation of acyl ureas having insecticidal activity.

**REEXAMINATION CERTIFICATE
ISSUED UNDER 35 U.S.C. 307**

THE PATENT IS HEREBY AMENDED AS
INDICATED BELOW.

Matter enclosed in heavy brackets [] appeared in the patent, but has been deleted and is no longer a part of the patent; matter printed in italics indicates additions made to the patent.

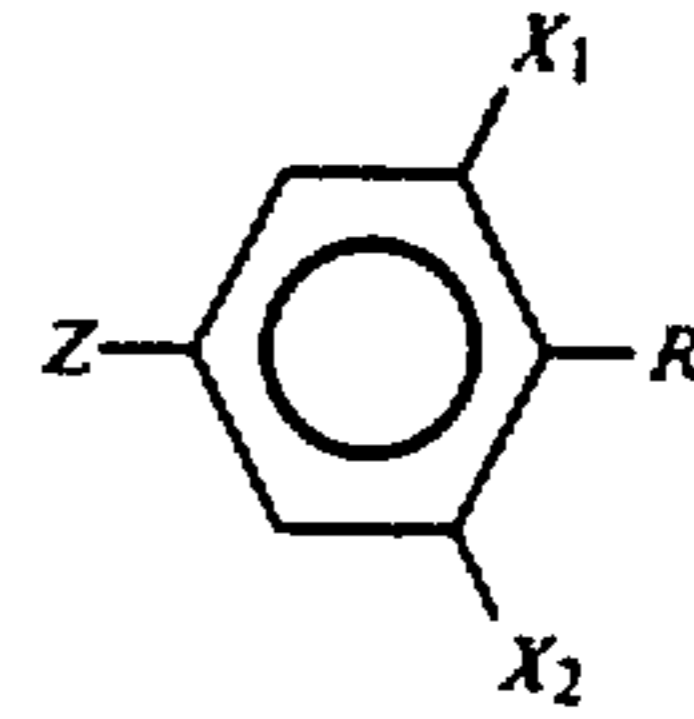
AS A RESULT OF REEXAMINATION, IT HAS
BEEN DETERMINED THAT:

Claim 1 is cancelled.

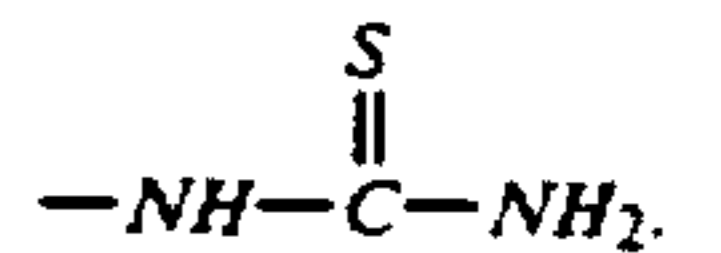
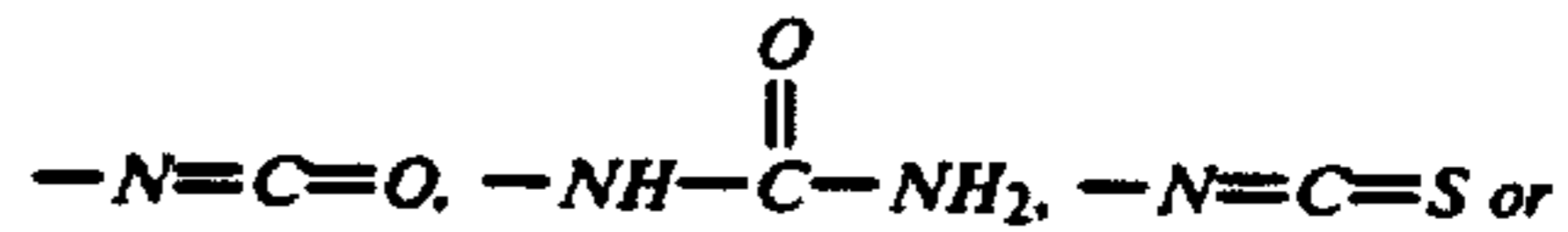
Claim 2 is determined to be patentable as amended.

Claims 3-5, dependent on an amended claim, are determined to be patentable.

2. [Compound of claim 1] *A compound having the formula*



wherein X₁ and X₂ are independently F, Cl or Br, [and] R is OCF₃, OCF₂CHCl₂, OCF₂CHF₂, OCF₂CHFCI or OCF₂CHFBr and Z is NH₂.



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